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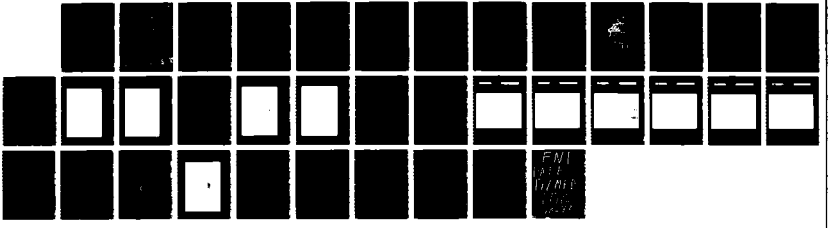

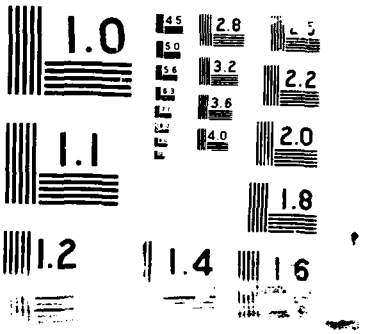
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The laser-induced fluorescence from Dy:YAG has been employed for the measurement of temperature on reacting and nonreacting surfaces. Point, line, and two-dimensional mapping of the surface temperature has been demonstrated utilizing the tripled output (355 nm) of a Nd:YAG laser. The temporal and spatial distribution of the temperature was recorded with a two-dimensional intensified imaging system. The technique has also been utilized for the measurement of the thermal depth profile of a plastic material undergoing rapid heating by a CO_2 laser.

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Foreword

This report was prepared by Larry P. Goss, Ph.D., Principal Investigator, and Michael E. Post and covers work performed under AFOSR Contract No. F49620-87-C-0040 during the period 17 February 1987 through 17 February 1988. The AFOSR contract monitor during this period was Julian M. Tishkoff, Ph.D.

TABLE OF CONTENTS

LIST OF ILLUSTRATIONS

LIST OF ILLUSTRATIONS (Continued)

Section I

INTRODUCTION

This report presents the first-year results of an experimental investigation into the use of Laser-Induced Fluorescence (LIF) as a diagnostic tool for remote sensing of the temperature of reacting and nonreacting surfaces. The goals of this two-year effort are (1) to extend LIF techniques to the measurement of two-dimensional surface temperatures, (2) to develop a probe for measuring thermal depth profiles for energetic materials, and (3) to apply fluorescence temperature techniques to the study of nonmetallic materials.

Efforts made during the first year of this contract toward achieving these goals are summarized in this report.

Section II

LABORATORY STUDIES

Line and Two-Dimensional Surface Studies

Effort during the first year of this program was directed mainly toward line and two-dimensional surface thermometry. The measurement technique which has been found to be the most effective for this purpose is Laser-Induced Fluorescence (LIF) of thermographic phosphors. This technique involves embedding small 10 - 100 μm sized thermographic-phosphor crystals in the surface to be studied. The LIF emission from these phosphors can be used to determine the temperature of the crystal and, thus, the surrounding surface. An ideal crystal for this purpose was found to be Dy:YAG; this crystal was employed for all the studies described in this report.

The temperature sensitivity of Dy:YAG crystals results from a "thermalization" process. When two energy levels of a rare-earth ion in a crystal field are separated by < 1000 cm^{-1}, the upper level typically will not fluoresce at low temperature due to extremely high multi-phonon relaxation rates which act to quench the closely spaced levels. Thus, at low temperatures no population buildup occurs in the upper energy level and, therefore, no fluorescence is observed. As the temperature increases, the upper energy level becomes more populated and the fluorescence from this level increases. Thus, by monitoring the increase in fluorescence of the upper level relative to the lower level, the temperature can be determined.

A prime example of this behavior is found in trivalent dysprosium doped at 3% yttrium-aluminum-garnet (Dy:YAG). The simplified energy diagram of Dy:YAG is shown in Fig. 1. Absorbed laser light (at 355 nm) can excite the Dy^{+3} ion into a high-energy state which radiatively and non-radiatively decays to the F-level. This level undergoes a fast thermal equilibrium which pumps a portion of its population into the nearby G-level. Fluorescence is then observed from both states. Figure 2 displays the fluorescence from both the F- and G-levels of Dy:YAG,

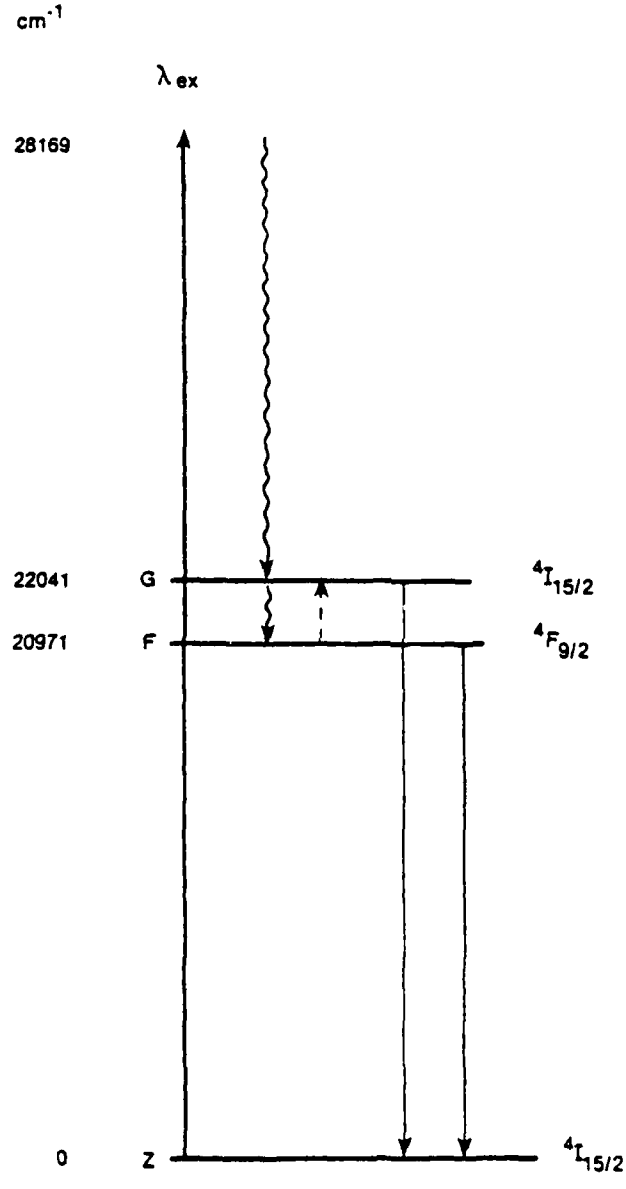

Figure 1. Simplified Energy Diagram of F- and G-Levels of Dy:YAG.

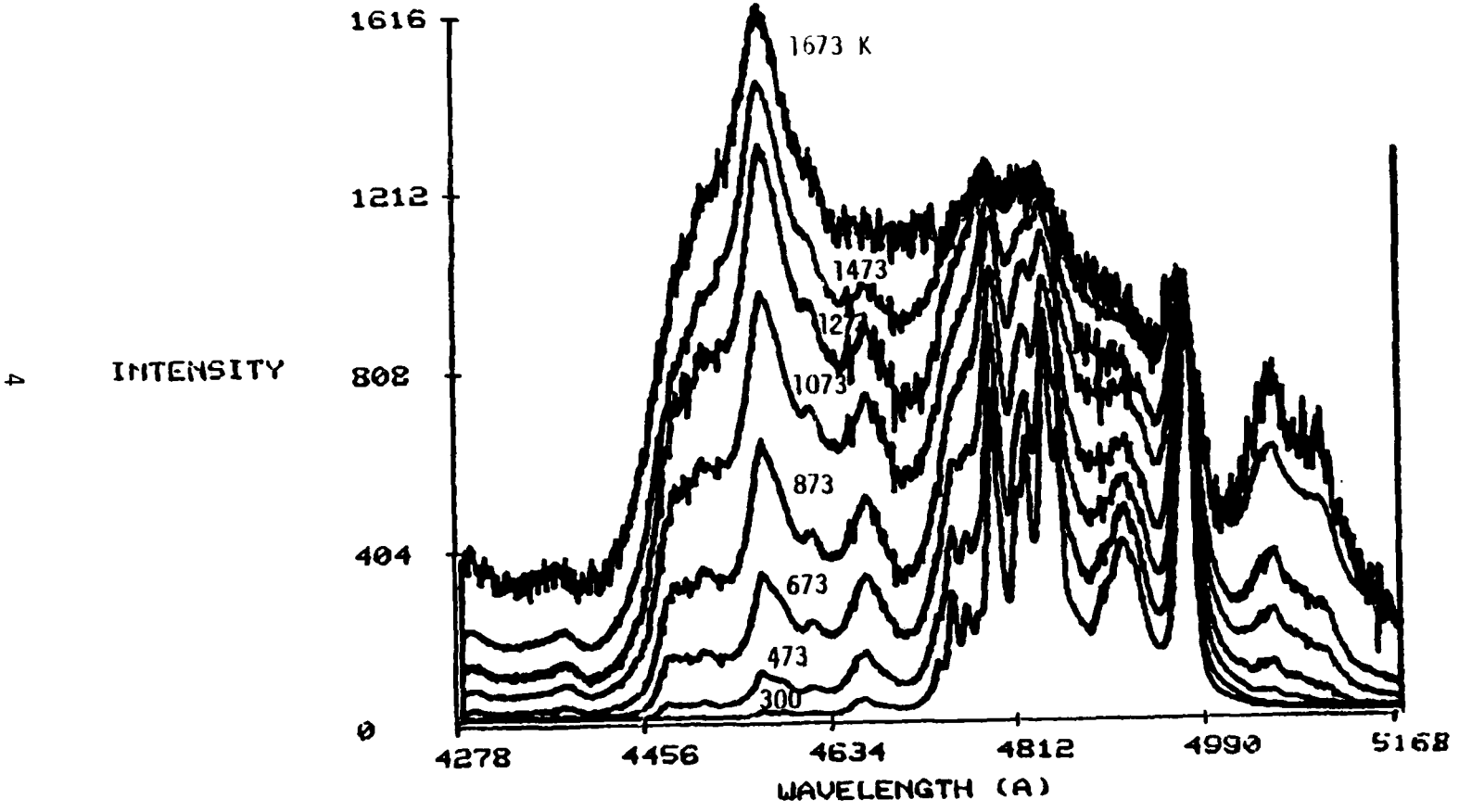

Figure 2. Fluorescence of F- and G-Levels of Dy:YAG as Function of Temperature.

including various Stark-shifted components, as a function of temperature. The intensity of the F-fluorescence level located at 496 nm is observed to remain relatively fixed as the temperature increases, while the intensity of the G-level component located at 467 nm increases dramatically with temperature. Because the 496-nm line is relatively insensitive to temperature, it can be used as an internal standard which allows the temperature to be determined from a ratio calculation rather than a more-difficult absolute measurement. The ratio of the 467 to 496 nm lines is shown in Fig. 3. A nonlinear curve observed for this ratio displays good temperature sensitivity up to 1800 K. The fluorescence from the F- and G-levels of Dy:YAG is extinguished at a temperature of $\sim$ 1800 K due to the large phonon quenching rates at this high temperature. The temperature range of the Dy:YAG crystals is more than adequate for monitoring the surface temperature of energetic materials.

One of the major advantages of employing LIF thermographic phosphors is the possibility of extending the technique to multi-dimensional measurements through the use of line and/or two-dimensional detectors (cameras). Simultaneous monitoring of the F- and G-fluorescence levels in this manner permits determination of the surface temperature at a point, along a line, or over the entire surface. The experimental arrangement employed for surface thermographic imaging is shown in Fig. 4. The main component of the system is a linear or two-dimensional intensified camera which records the fluorescence from the surface under study. In the case of line measurements, the detector employed is a Tracor-Northern TN1710 1024-element diode array. For two-dimensional measurements the Tracor-Northern is replaced with an intensified CCD camera system manufactured by Photometrics, Ltd. In both cases the fluorescence from the surface is excited by the tripled output of a Quanta-Ray Nd:YAG laser. The intensified detector is gated with a 10-μs pulse which is an aid in discriminating against unwanted background radiation from the surface. This feature is very important at elevated temperatures where the natural blackbody emission from the surface and surroundings can be particularly large. The fluorescence from the surface is collimated by an F/6 lens, split into two paths, and directed through 496- and 467-nm filters corresponding to the F- and G-fluorescence levels, respectively. The two paths are then imaged side by side on the linear or CCD camera. The

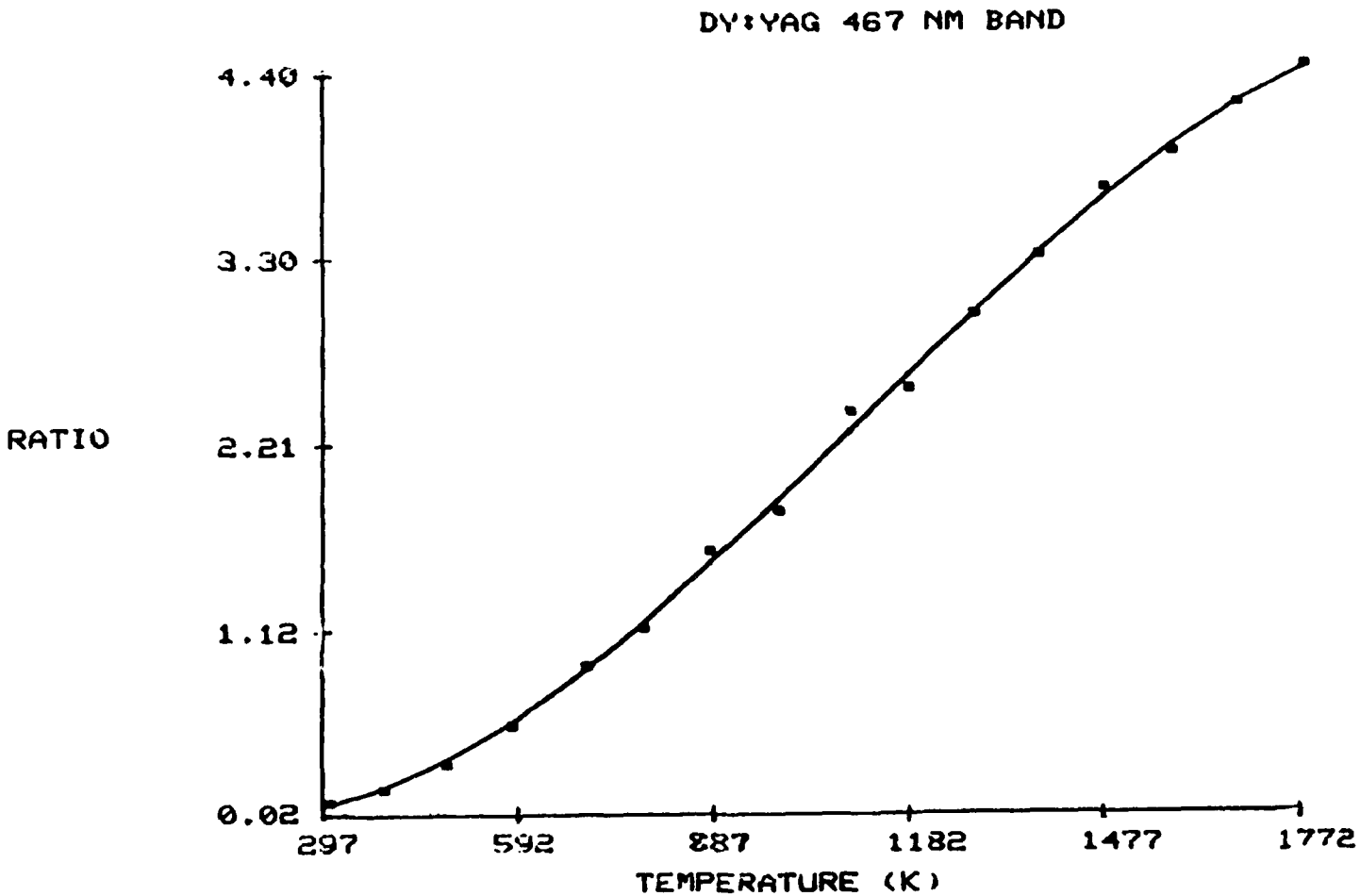

Figure 3. Variation of Relative Intensity of 467-nm G-Level Stark Component to 496-nm F-Level of Dy:YAG as Function of Temperature.

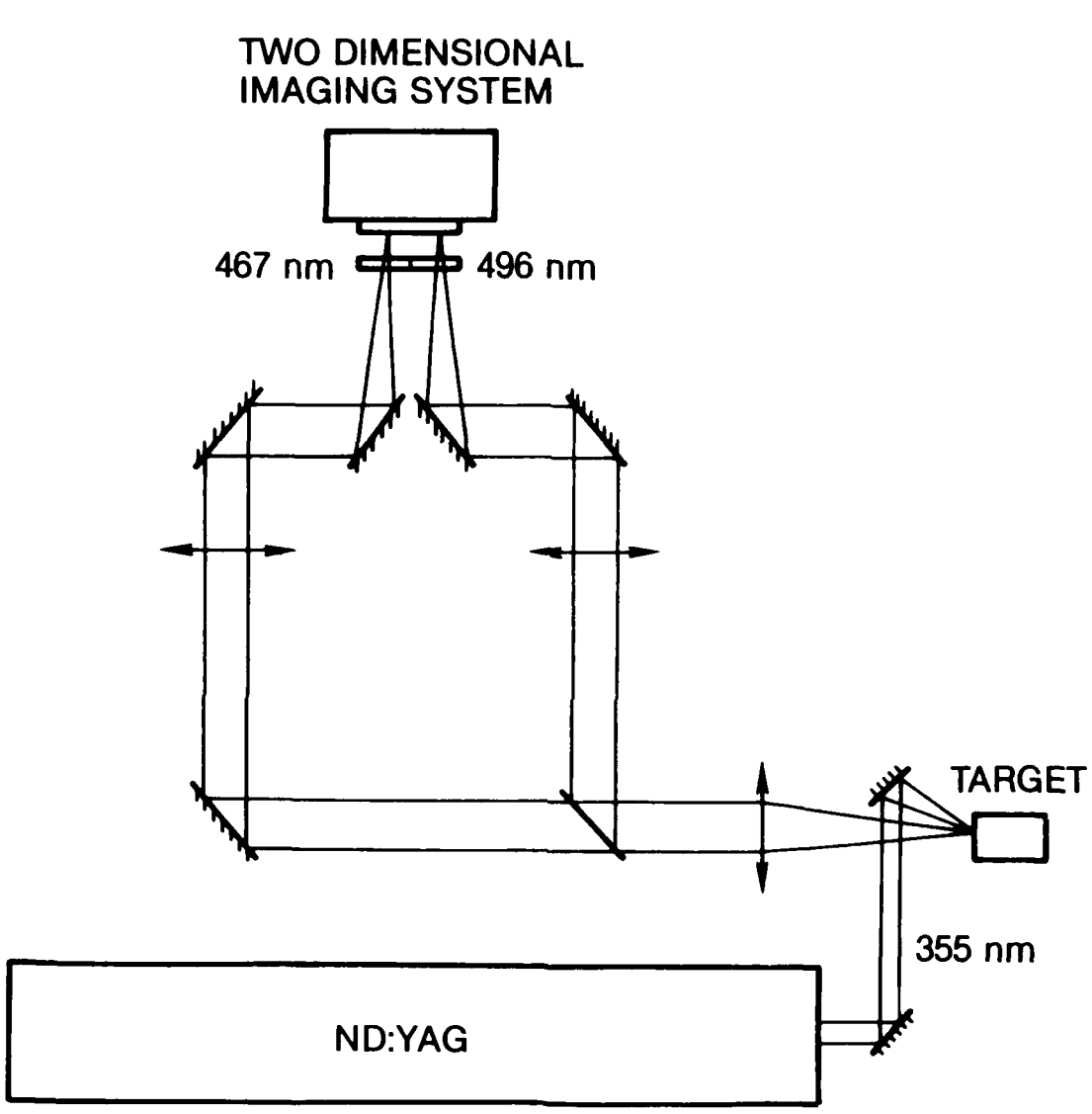

Figure 4. Experimental Arrangement Employed for Surface Thermographic Imaging.

video signal from the detector is digitized, stored in temporary memory, and subsequently sent to a ModComp mini-computer for analysis. Image analysis consists of ratioing the F- and G-fluorescence signals and converting the resulting relative intensity to a temperature with the aid of a calibration curve established using a platinum-coiled oven. The surface temperature can then be displayed as a function of time and/or space. The Tracor-Northern linear-array detector is capable of 30-Hz operation and, thus, has been employed for time-dependent temperature studies. Since the Photometrics CCD has a much lower data-acquisition rate, it has been employed for precise spatial measurements.

The linear-time-resolved imaging effort has been concentrated on inert ceramic and plastic surfaces. The various heat sources used to increase the surface temperature of the test specimen range from embedded Nichrome wires to CO_2 laser excitation. The Dy:YAG crystal was bonded to the ceramic surfaces with a high-temperature ceramic adhesive. The crystals were intermixed with the plastic materials before curing, which allowed the surface to be replenished as it was being eroded by high-temperature excitation.

The embedded-Nichrome-wire excitation in a ceramic is shown in Fig. 5. This color contour depicts the change in surface temperature along a line located perpendicular to the embedded wire. The temperature is displayed as a function of time as electrical current is allowed to flow through the Nichrome wire and heat the surface. After $\sim$ 4.5 s, the electrical current is discontinued and the surface allowed to cool. From this figure the lateral spread in temperature as well as the cooling decay time can be determined.

Next, the ceramic surface was heated at a much higher rate by a CO_2 laser. The color contour of the temperature along a line intercepting the focal spot of the laser is shown in Fig. 6. The risetime in temperature is much faster with the CO_2 excitation than in the Nichrome-wire study. The temperature at the focal spot is also much higher than in the resistance-heating experiment. The CO_2 laser excitation began at $\sim$ 4 s and ended at 10.5 s. The temperature then abruptly decayed, eventually reaching room temperature.

Figure 5. Color Contour Depicting Change in Surface Temperature Along Line Located Perpendicular to Embedded Wire.

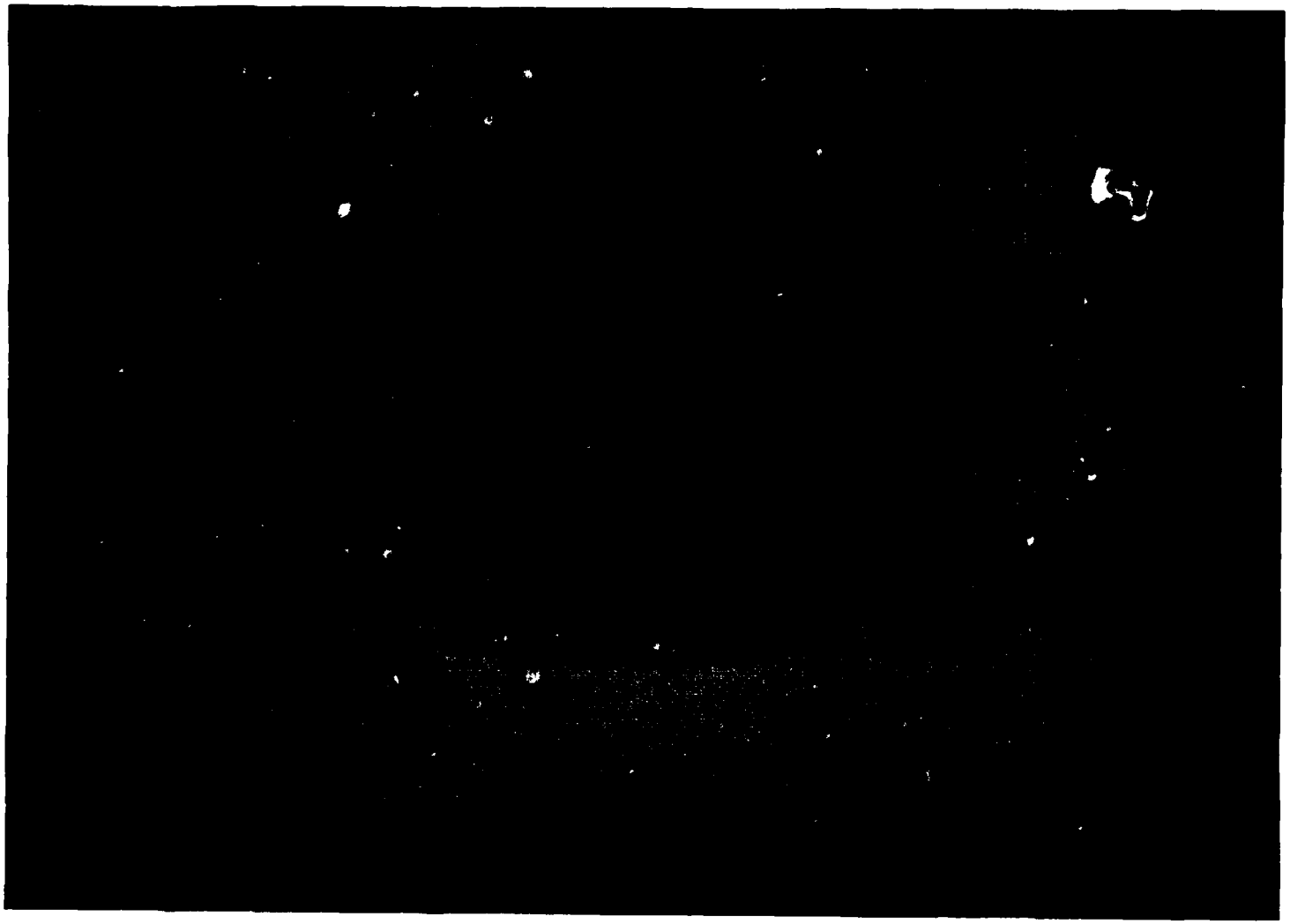

Figure 6. Color Contour of Temperature Along Line Intercepting Focal Spot of Laser.

The results of the first experiment with a plastic target are shown in Fig. 7. The surface of the plastic in this case was being heated with a propane torch which was covering the entire measurement area. The torch was applied to the surface at approximately the 4.5-s mark, and the temperature at the surface increased to $\sim$ 550 K.

Finally, the CO_2 laser was used to heat the plastic surface, and the results are shown in Fig. 8. The color contour shows the much higher surface temperature which was achieved by CO_2 excitation, with a maximum temperature over 700 K. The temperature decay after termination of the CO_2 excitation is shown beginning at $\sim$ 15.5 s. The plastic reached a lower temperature than the ceramic. This is partially due to the energy used in eroding and melting the plastic.

The majority of the effort with the Photometrics CCD camera was expended in developing the software and hardware needed to synchronize the Nd:YAG laser and the camera. The acquisition and analysis software were written in Forth, and a custom interface was developed for downloading the image to the ModComp computer. Preliminary measurements have been made on a nonreacting, doped ceramic surface. Figure 9 is a sketch of the ceramic block used for this initial experiment. The test block consisted of a cylindrical piece of ceramic with two parallel Nichrome wires suspended above its surface. The Nichrome wires in this case were coated with a ceramic adhesive and doped with Dy:YAG crystals. A current was allowed to flow through the wire to produce a local heat source which was monitored with the thermographic phosphors. The experimental arrangement is the same as that shown in Fig. 4, with the Photometrics CCD camera being used as the two-dimensional imaging system.

The series of two-dimensional images which were obtained during a heat-up and cool-down cycle of the Nichrome wires is shown in Figs. 10 - 15. Each image consists of two regions, showing the fluorescence from the parallel wires. One-half of each figure corresponds to the 467-nm fluorescence signal, while the other half corresponds to the 496-nm fluorescence signal. As the temperature of the wires increases, there is a corresponding increase in the intensity of the 467-nm component, as shown in Figs. 10 - 15. During this experiment images were

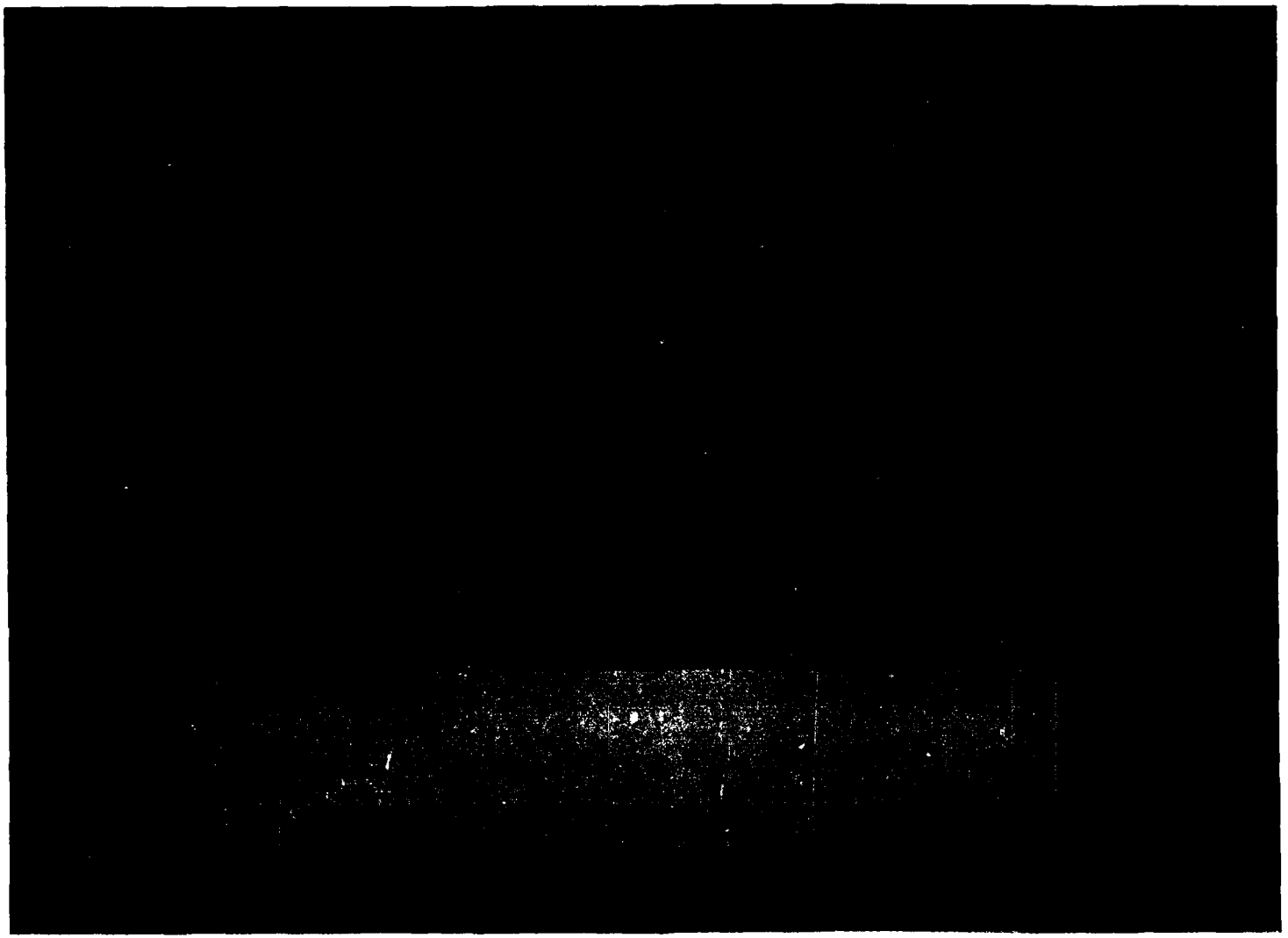

Figure 7. Color Contour Showing Results of Heating Plastic Surface with Propane Torch.

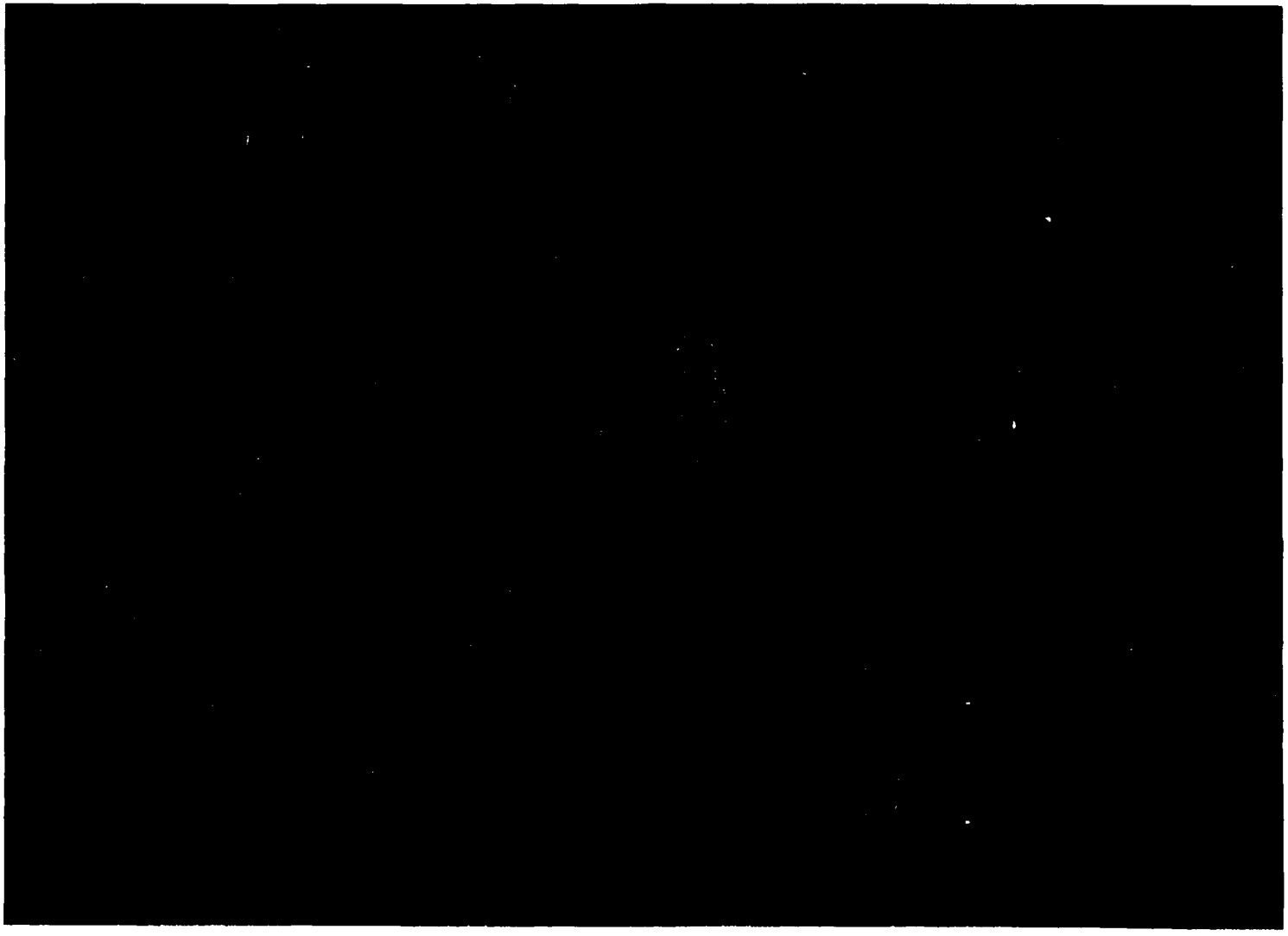

Figure 8. Color Contour Showing Results of Heating Plastic Surface with CO_2 Laser.

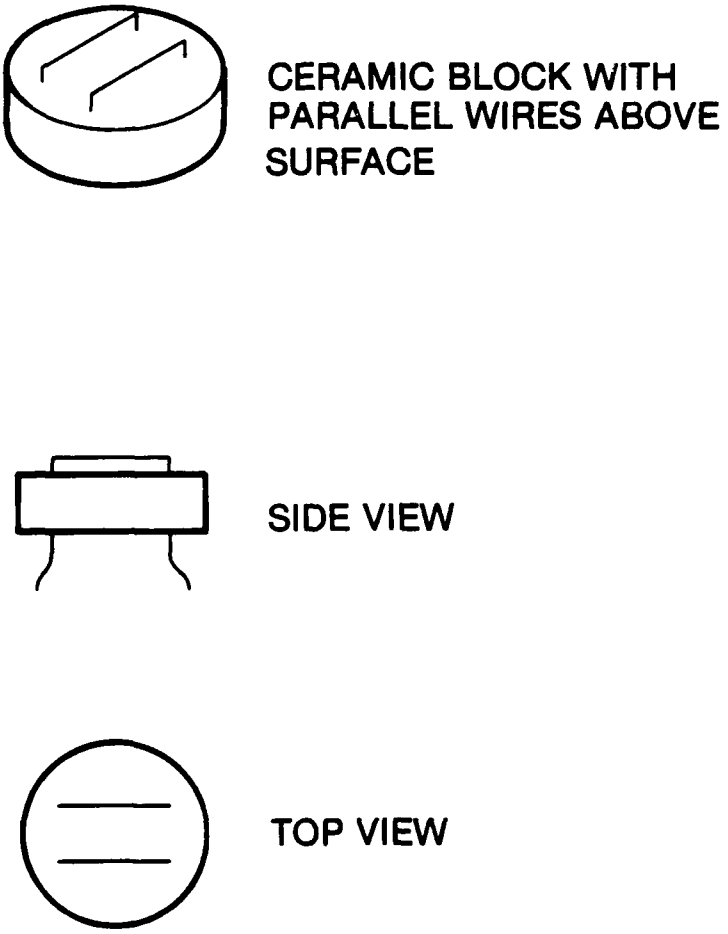

Figure 9. Sketch of Ceramic Block Used for Initial Experiment.

taken at 1-s intervals. For 4 s no current was applied; then the Nichrome wires were heated for 8 s, after which the current was turned off and the cooling cycle observed for 20 s. Figure 10 displays the fluorescence image prior to the heating cycle. Figures 11 - 12 show the increase in the 467-nm fluorescence during a heating cycle. After $\sim$ 8 s the current to the wires is discontinued and the temperature begins to drop, as shown in Figs. 13 - 15. Figure 16 is a plot of a single spatial point for both the 467-nm and the 496-nm fluorescence during the heat-up and cool-down cycles. The maximum temperature of 500 K is reached in $\sim$ 8 s.

The CCD camera interface and software development are near completion; therefore, studies with the two-dimensional camera can proceed as planned. These include determination of surface-roughness effects, nonuniformity of the dopant, minimal dopant levels, and detector sensitivity. Preliminary efforts are to be carried out on plastic materials, with final efforts to be directed toward doping and studying energetic materials.

Thermal Depth Probe Studies

Because of the importance of surface temperature and thermal penetration depth in controlling the reaction rate of energetic materials, an effort was undertaken to apply the LIF-thermographic-phosphor technique to the problem of developing a thermal depth probe. The classical approach to this measurement involves embedding small thermocouples into the sample and allowing the sample to burn through the thermocouple location. This approach requires the use of small fragile thermocouples which are destroyed during the measurement process. The thermocouples in practice must be much smaller than the desired spatial resolution in order to minimize thermal conduction along the lead wires. Problems of determining the exact location of the surface during the burn-through must also be addressed.

One approach in which the thermographic phosphors can be utilized in determining the thermal profile involves trimming the edge of the material to be studied to provide a flat surface and imaging the

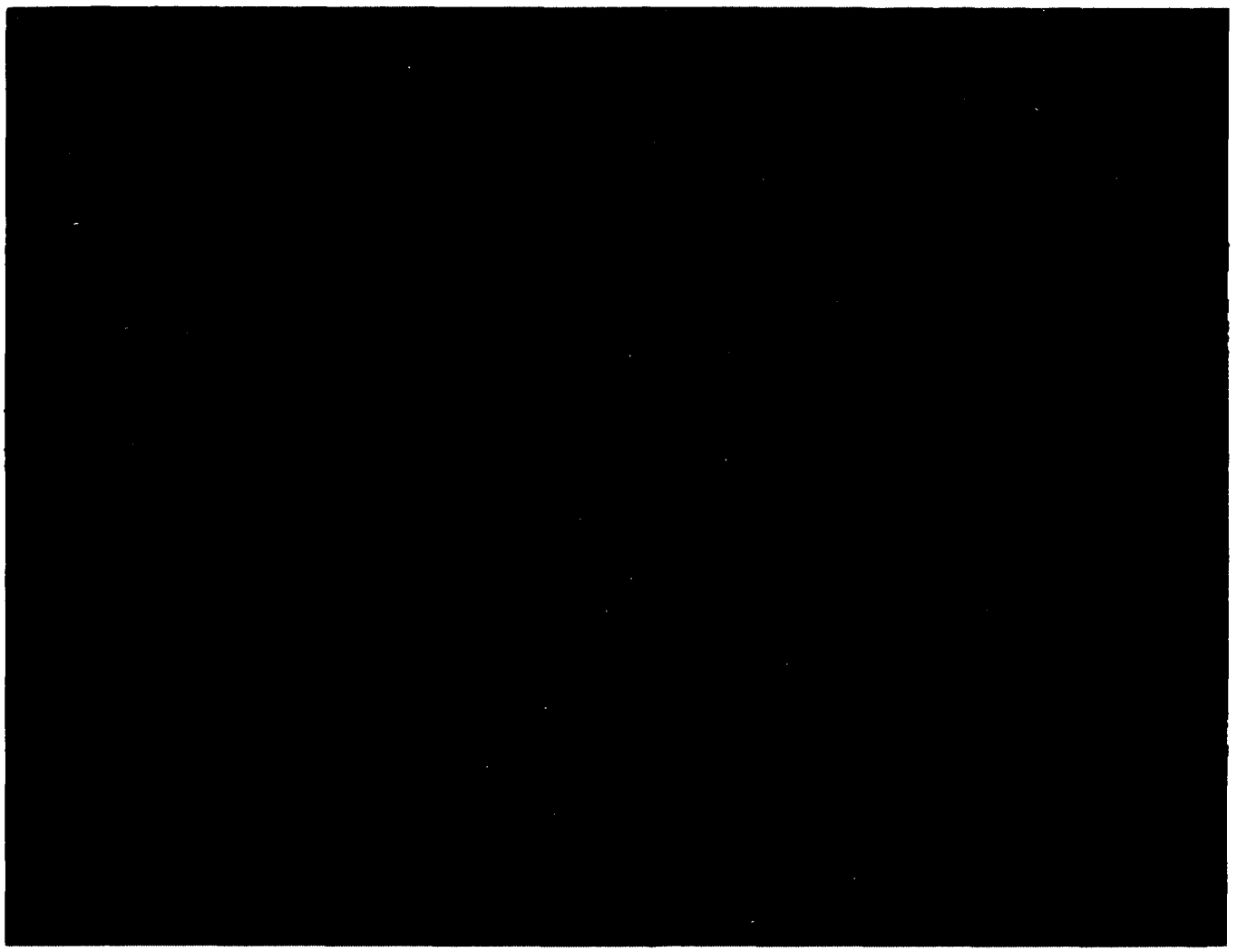

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Figure 10. Fluorescence Image of Nichrome Wires Prior to Heating Cycle (Left - 496-nm Signal; Right - 467-nm Signal).

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Figure 11. Increase in 467-nm Fluorescence During Heating Cycle (2 s after current was turned on).

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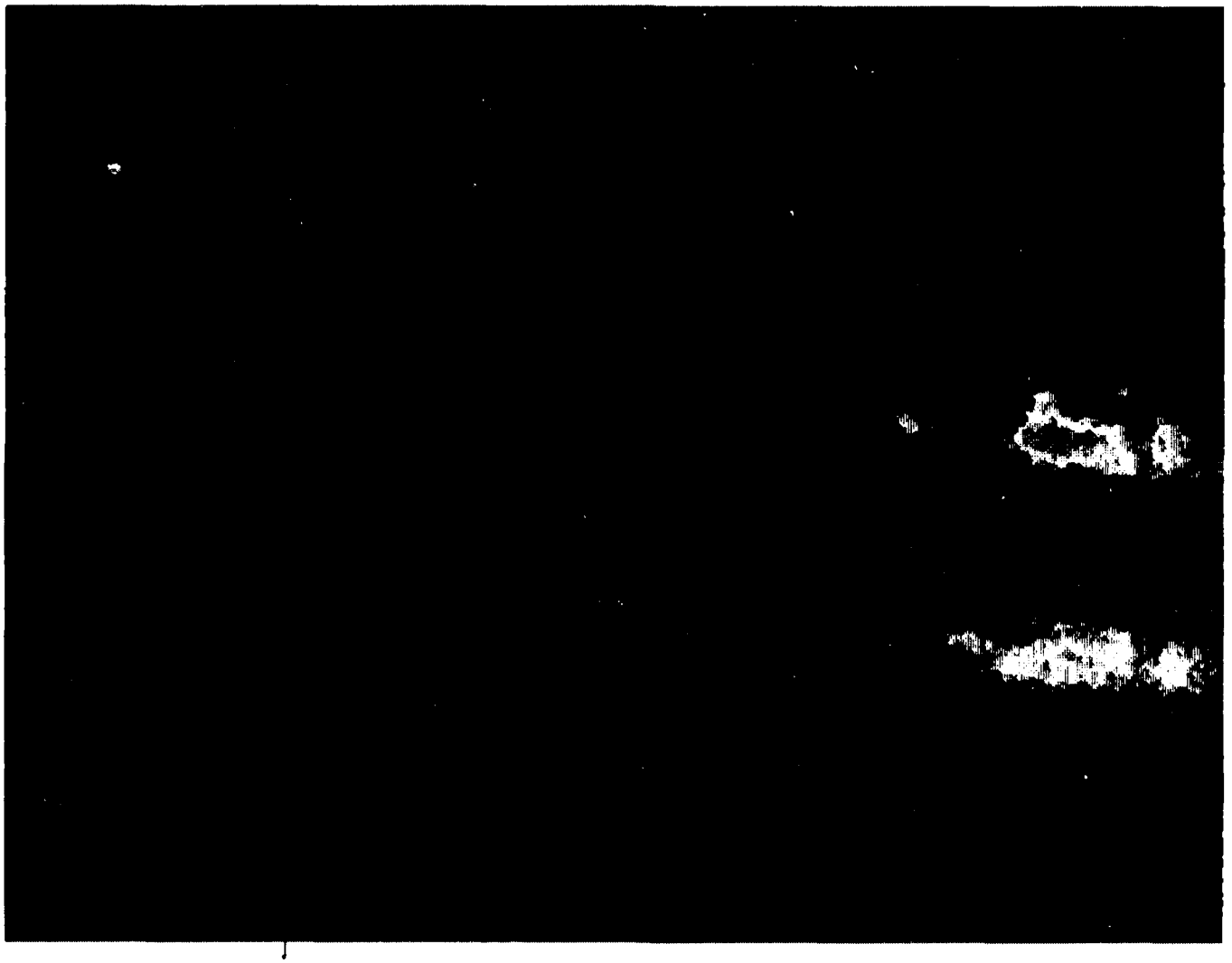

Figure 12. Further Increase in 467-nm Fluorescence During Heating (8 s after current was turned on).

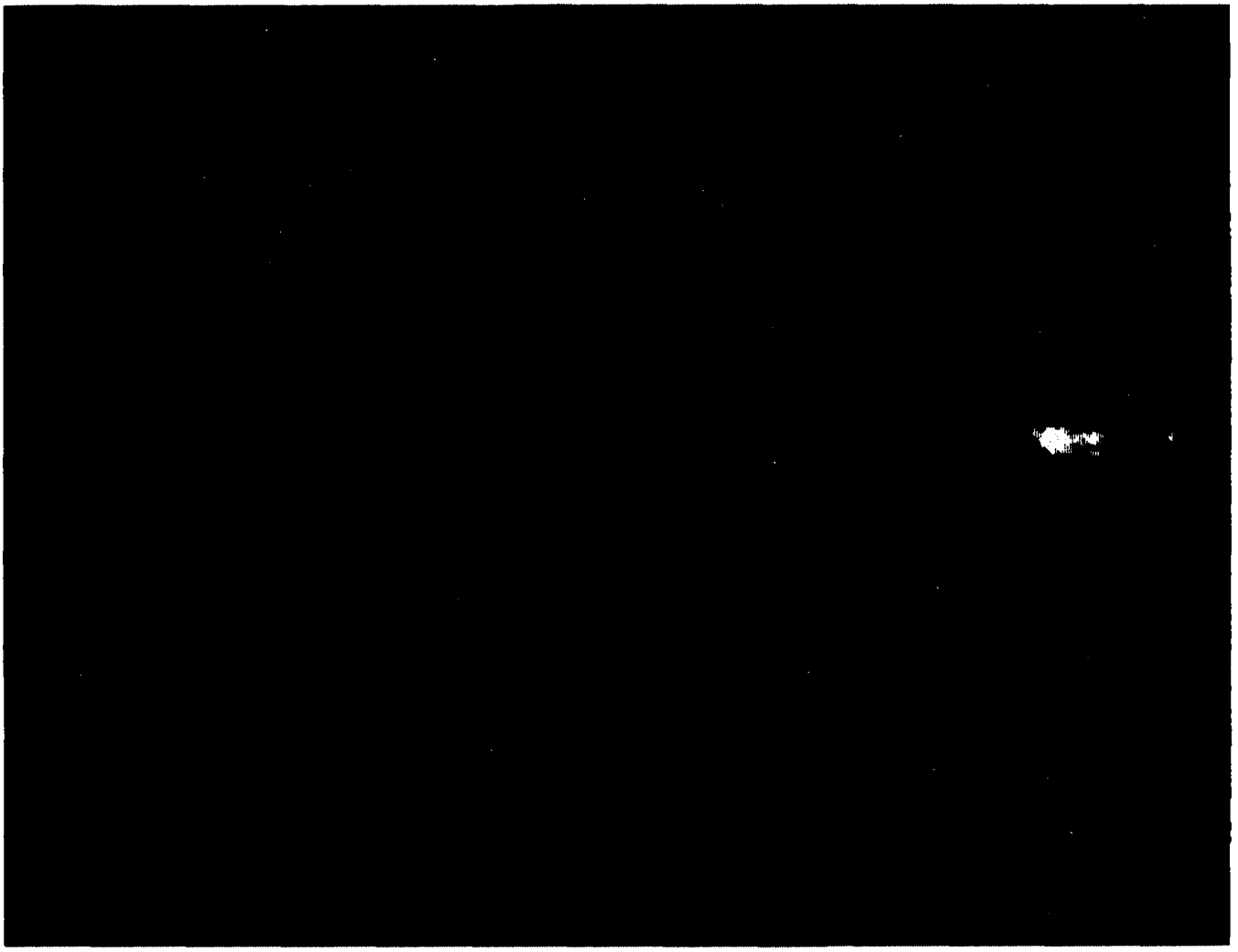

Figure 13. Decrease in 467-nm Fluorescence When Current Discontinued (6 s after current was turned off).

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Figure 14. Further Decrease in 467-nm Fluorescence When Current Discontinued (14 s after current was turned off).

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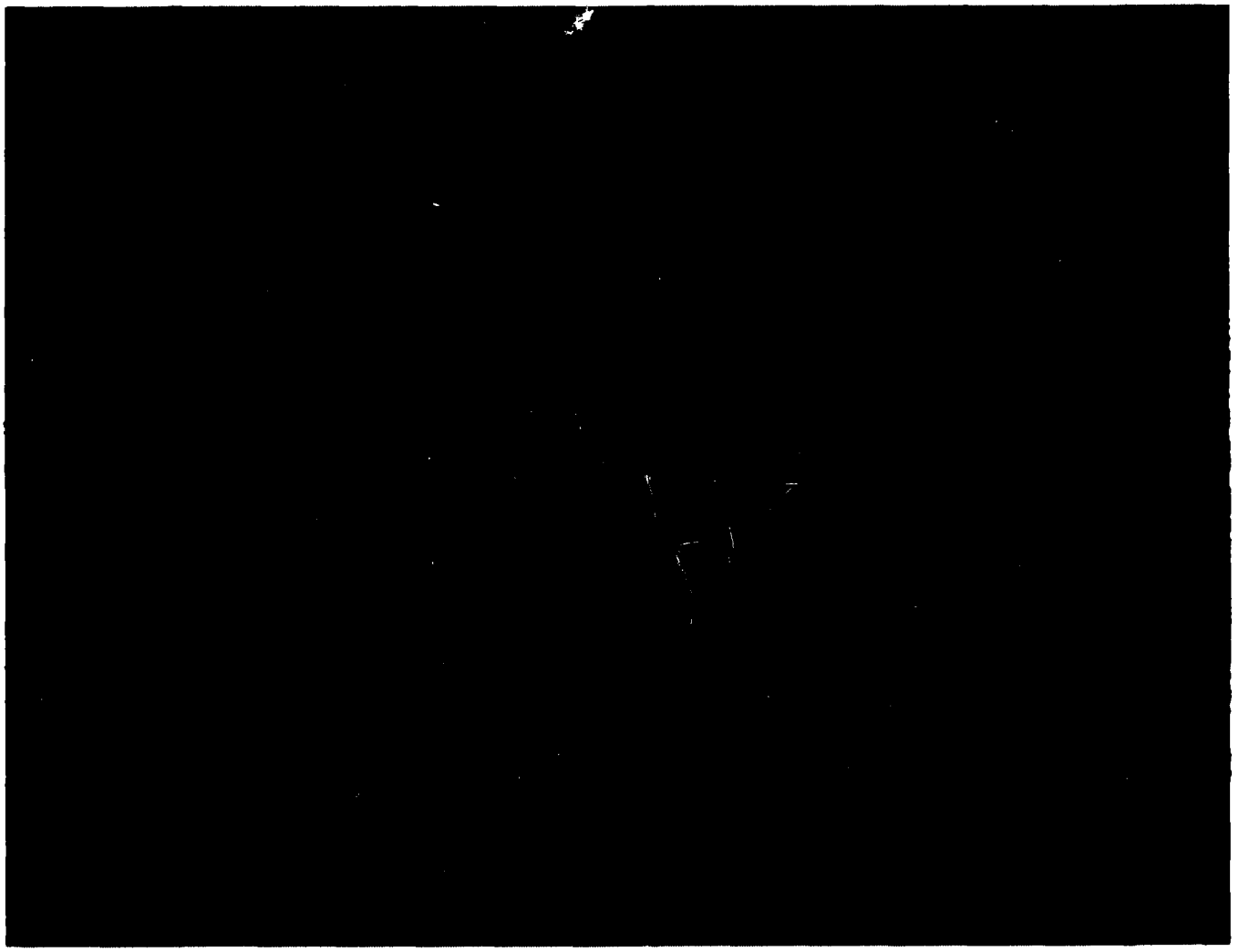

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Figure 15. Further Decrease in 467-nm Fluorescence When Current Discontinued (20 s after current was turned off).

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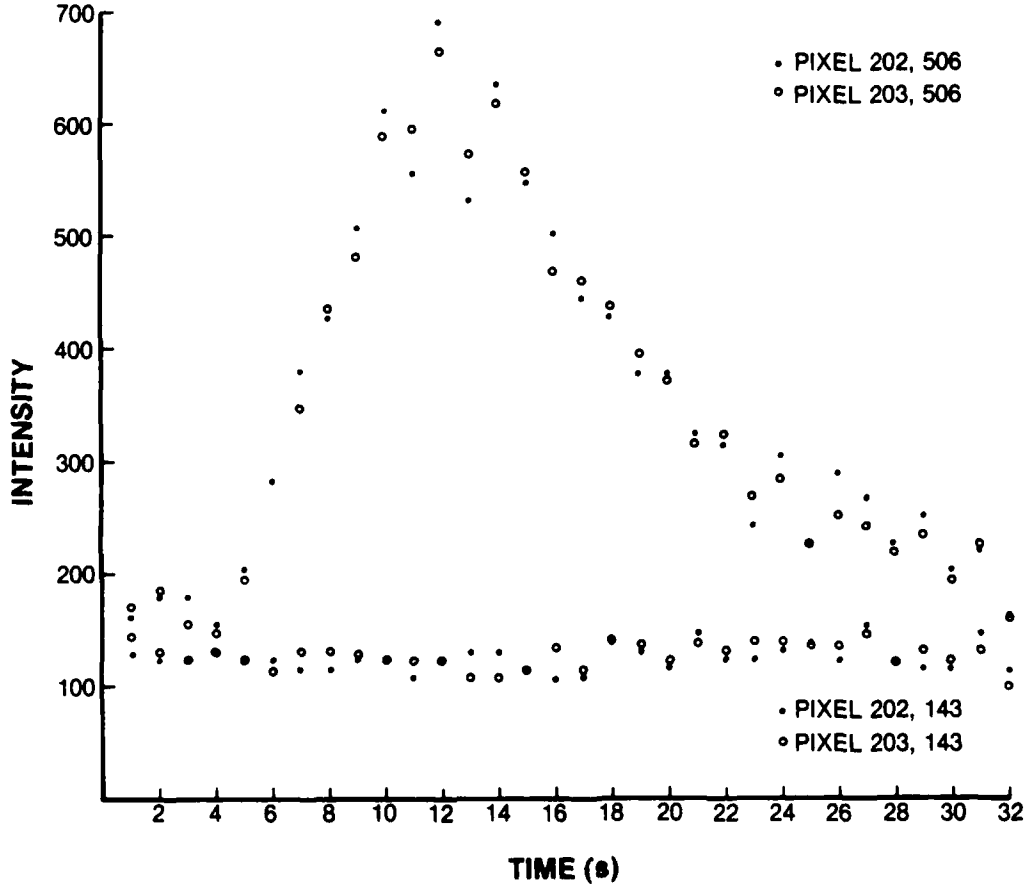

Figure 16. Plot of Single Spatial Point for 467-nm (Pixels 202, 506 and 203, 506) and 496-nm (Pixels 202, 143 and 203, 143) Fluorescence During Heat-Up and Cool-Down Cycles.

fluorescence from this side during the combustion process. This allows the entire depth profile to be monitored as a function of time--two dimensionally rather than at a single point. This approach is demonstrated in Fig. 17. In this case a CO_2 laser was used as the heat source and directed along the edge of a plastic target. The edge was illuminated by the tripled Nd:YAG beam (355 nm) and the fluorescence (temperature) recorded over a period of time. As the plastic heated and eroded, the CO_2 beam penetrated further along the edge of the sample. The temporal history of the temperature profile was monitored while this erosion was taking place. The observed thermal depth profile is displayed in Fig. 18. This figure depicts the gradual buildup of the surface temperature, the plateau at the maximum surface temperature, and the penetration of the heat into the plastic as a function of depth and time.

Alternative approaches which are being considered for this task include the development of a fiber-optic probe which can be embedded into the material in the same manner as a thermocouple. The fiber will be used to transmit the exciting UV laser light as well as transmit the resulting fluorescence signal from the doped tip. The probe design which is being considered is shown in Fig. 19. A beam splitter will be used to separate the UV laser from the visible fluorescence. The advantage of the probe configuration is that it is single ended and can be used in limited-optical-access situations.

Several sapphire and quartz optical fibers have been obtained, and development of the probe has begun. Methods for bonding the Dy:YAG crystals to the probe are presently being investigated.

Nonmetallic Materials and Surface Bonding Studies

The objective of this final portion of the program is to identify and study techniques for bonding the Dy:YAG crystal to surfaces of various nonmetallics which are of interest to the U. S. Air Force. Techniques that result in a relatively thin layer of crystals which are in good thermal contact with the surface are of primary interest. Techniques such as sputtering, electron-beam deposition, flame spray, and plasma

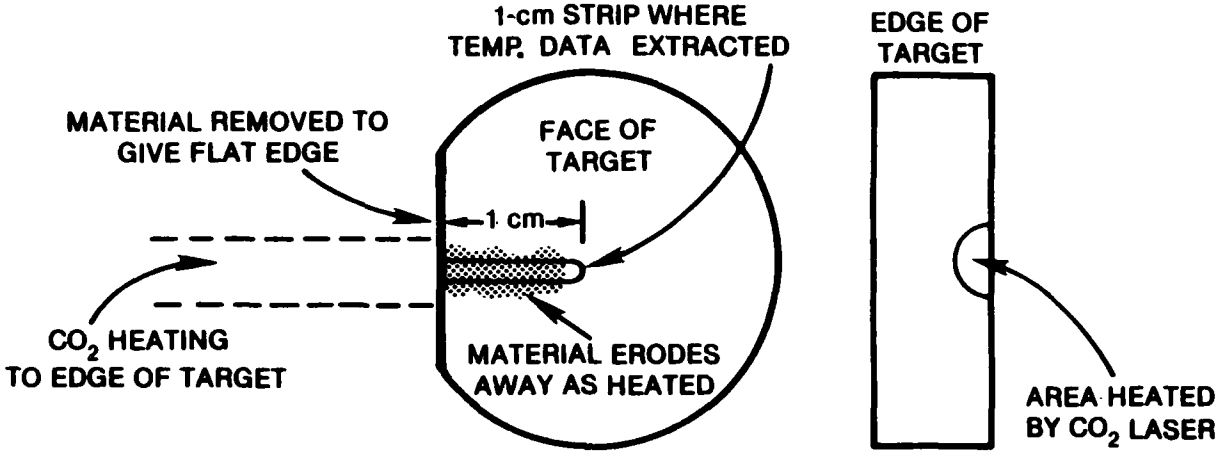

Figure 17. Schematic Diagram of Method for Obtaining Thermal Depth Profile of Energetic Material.

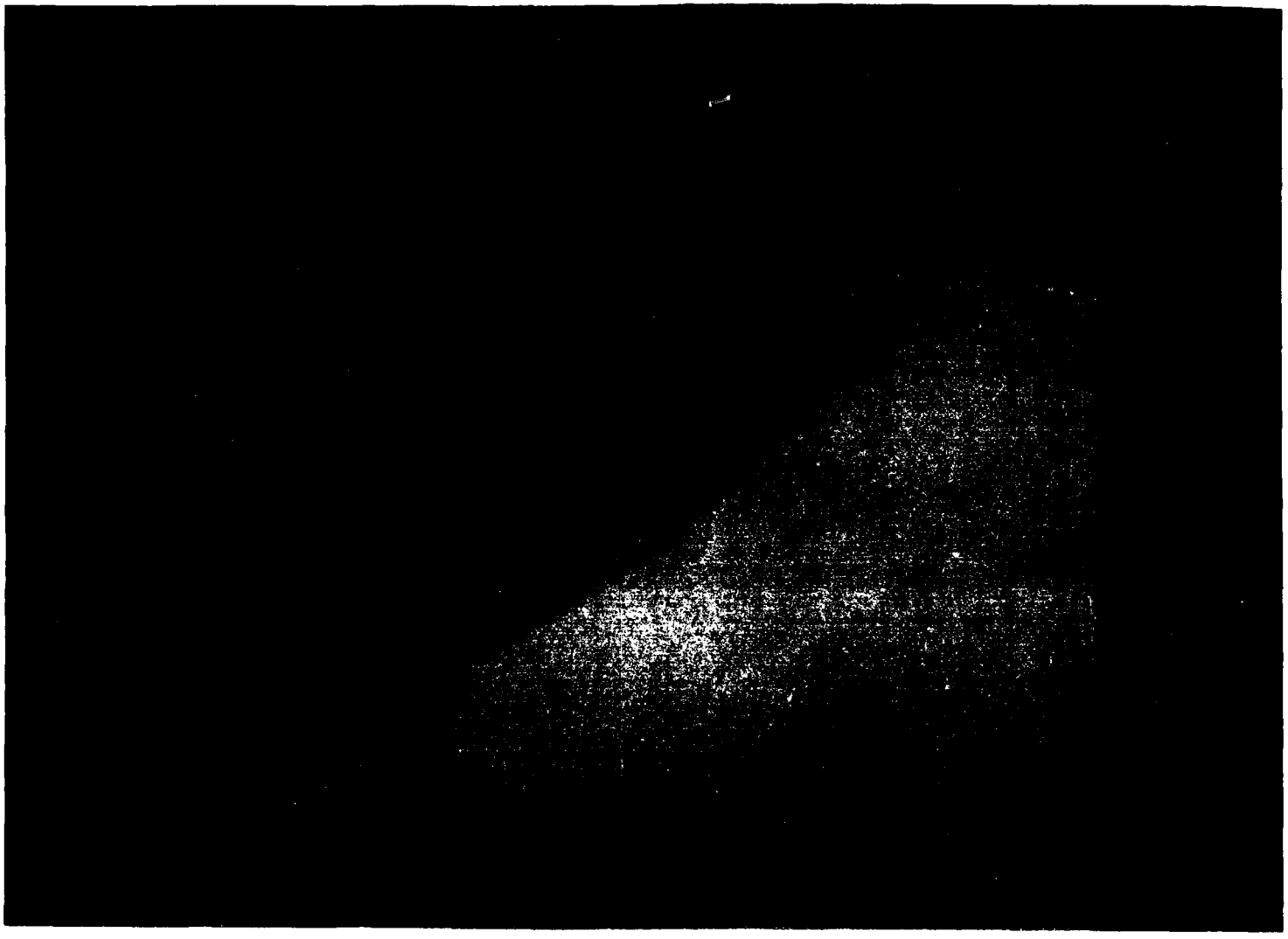

Figure 18. Color Contour Displaying Thermal Depth Profile of Plastic Target Heated with cw CO_2 Laser ~ 30 W.

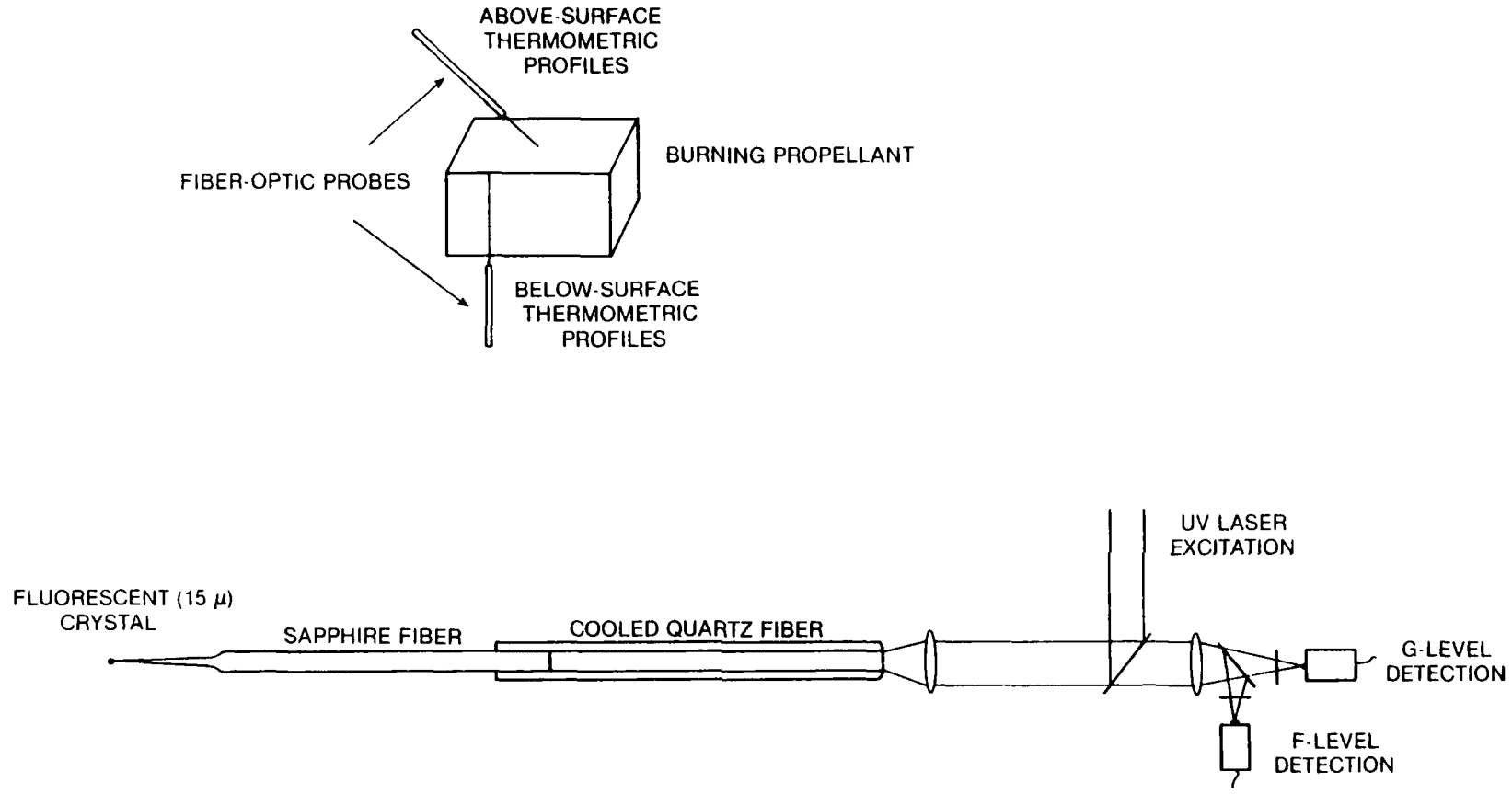

Figure 19. Proposed Fiber-Optic Probe for Thermal Depth Profiling.

spray have been considered. Dy:YAG crystals can be melted and recrystallized without altering the properties which make them desirable for LIF. Sputtering and electron-beam deposition methods are generally used for metals, and most of the readily available equipment which makes use of these methods can only accommodate small objects (2 - 5 cm^2). Flame-spray is more portable and applicable to objects of diverse geometries, but it has a lower impact velocity than plasma spray. Plasma spraying allows a strong bond to be formed, and bonding to ceramic materials is especially effective.

In addition to thermal bonding techniques, methods have also been considered which may employ high-temperature adhesives. These materials include silicates, ceramics, and epoxies. Two-dimensional imaging of heated, inert material was demonstrated by bonding Dy:YAG crystals to the surface of a high-temperature ceramic adhesive.

Plasma spray will be studied for crystal deposition on nonmetallic surfaces. Bonding characteristics as well as thermal fatigue studies will be conducted on serveral nonmetallic materials of importance to the U. S. Air Force. Thermometric imaging will then be conducted under simulated practical conditions.

Section III

PRESENTATIONS AND PUBLICATIONS

The following presentation has resulted from the research performed during this reporting period:

"Surface Thermometry of Energetic Materials by Laser-Induced Fluorescence," L. P. Goss and M. E. Post, To be presented at the AFOSR/ONR Propulsion Contractors' Meeting, 13-17 June 1988, Monrovia, California.

Section IV

PROFESSIONAL PERSONNEL ASSOCIATED WITH RESEARCH EFFORT

During this reporting period the following professional personnel have been associated with the research effort:

Larry P. Goss, Ph.D.
Principal Investigator

Arthur A. Smith
Project Physicist

Michael E. Post
Research Physicist

Section V

SUMMARY

Work is in progress on all three tasks of this program, with the most significant accomplishment of the first year being the demonstration of the two-dimensional thermal imaging of heated inert surfaces. Thermal depth profiling has been conducted with a linear array, while the development of a fiber-optic thermal probe is in progress. Preliminary efforts on bonding techniques of the Dy:YAG crystals were initiated, with plasma spray holding the most promise for nonmetallics. Future efforts on the project will be directed toward two-dimensional measurements on energetic materials, completion of thermal-probe development and applications, and identification of optimum bonding methods for nonmetallic materials.

Respectfully submitted,

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